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The production of platinum-coated silicate nanoparticle aggregates for use in hypervelocity impact experiments

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Abstract

We present a method for producing metal-coated low-density ($\sim 1600 \text{ kg m}^{-3}$) aggregate silicate dust particles for use in hypervelocity impact (HVI) experiments. Particles fabricated using the method are shown to have charged and electrostatically accelerated in the Max Planck Institut für Kernphysik (MPI-K) 2 MV Van de Graaff accelerator, allowing the production of impact ionization mass spectra of silicate particles (impacting at velocities ranging from $<4 \text{ km s}^{-1}$ to $>30 \text{ km s}^{-1}$, corresponding to sizes of $>1 \text{ }\mu\text{m}$ to $<0.1 \text{ }\mu\text{m}$) using the Large Area Mass Analyser (LAMA) instrument, designed for cosmic dust detection in space. Potential uses for the coated grains, such as in the calibration of aerogel targets similar to those used on the Stardust spacecraft, are also discussed.

Key words: Interplanetary dust, Hypervelocity impact, Interstellar dust

1. Introduction

The composition of interstellar and interplanetary dust (cosmic dust) can reveal a great deal about the various sources of the dust particles. Thorough reviews regarding the composition and sources of interstellar and interplanetary dust can be found in, amongst others: Grün et al. (2009), Mann et al. (2006), Baggaley (2004), Draine (2003), Bradley (2003), Sekanina et al. (2001), Dorschner (2001) and Jessberger et al. (2001), although for clarity we present a brief summary of the main dust sources and the resulting dust compositions here.

Originating from outside the solar system, interstellar dust (ISD) is generated by AGB stars (Whittet, 1989) and, to a lesser extent, by novae, supernovae and Wolf-Rayet stars (e.g. Zhukovska et al., 2008). UV, visible and IR spectral features (e.g. Chiar & Tielens, 2006; Dorschner, 2001; Bradley et al., 1999; Begemann et al., 1997), together with modelling (Zhukovska et al., 2008) and laboratory analysis of interstellar particles returned by spacecraft (e.g. Brownlee et al., 2006) or trapped in meteorites (e.g. Hoppe et al., 2001) indicate that ISD has compositions which can include silicates, metal oxides, silicon carbide, amorphous carbon, polyaromatic hydrocarbons (PAHs) and volatile species.

Interplanetary dust (IDP) derives from sources within the solar system that include: comets (dust ejection via outgassing), asteroids (collisions and dust impact ejection), atmosphereless planetary satellites (rare collisions, possible outgassing/cryovolcanism, and frequent dust impact ejection), Edgeworth-Kuiper Belt objects (collisions, dust impact ejection and possibly outgassing), the Jovian and Saturnian systems (electromagnetically accelerated

stream particles) and, in the vicinity of the Earth, mankind itself (primarily rocket propellant residues).

The composition of cometary dust has been investigated both via in-situ impact ionisation mass spectrometry (PIA, e.g. Kissel, 1986; Jessberger & Kissel, 1991; PUMA, e.g. Kissel & Krueger, 1987; Dikov et al., 1989; and Stardust's CIDA, e.g. Kissel et al., 2004), via remote sensing (reflectance spectra from, for example, the recent Deep Impact mission (Lisse et al., 2006)) and via laboratory analysis of returned samples (e.g. Flynn, 2006, Flynn, 2008). The dominant materials in cometary dust are silicates, with contributions from carbonaceous materials (CHON particles), sulphides, metals, carbonates and oxides (Sekanina et al., 2001; Flynn, 2006; Flynn, 2008).

Dust with an origin on atmosphereless bodies (asteroids, planets, planetary moons, Edgeworth-Kuiper Belt Objects) should have a composition that reflects the surface composition of the source body (or, if generated by catastrophic collisions, the bulk composition). For the asteroids, both reflectance spectra (e.g. Gaffey et al., 1993 or, for example in the case of asteroid families, Mothé-Diniz et al., 2005) and terrestrial analysis of meteorites (e.g. Gaffey, 1976; Bradley & Brownlee, 1991; Bradley et al., 1999) indicate that silicates are, again, the dominant material, with contributions from metallic iron and nickel, and sulphides and oxides. Reflectance spectra (and, in the case of the Moon, direct laboratory analysis of the surface material e.g. Maxwell et al., 1970) of larger planetary satellites (or planets) (Carlson et al., 1996; Cruikshank et al., 2005 for example) indicate that dust generated from these objects will contain varying levels of ices, organics, salts and silicates (Europa, e.g. Fanale et al., 1999; Enceladus, e.g. Brown et al., 2006; Dione, e.g. Clark et al.,

2008; the Moon and Mercury, e.g. Warell et al., 2006) depending on the particular source body, and the region of the source from which the dust was ejected. In rare cases, such as that of Enceladus, the dust particle composition has been directly measured (Hillier et al., 2007b; Postberg et al., 2008; Postberg et al., 2009a), confirming and expanding on the results from remote observations.

Stream particles – nanometre scale dust that charges and accelerates within the magnetosphere of their source giant planetary system before being ejected into interplanetary space, carries the compositional fingerprint of the source region (or regions) within the planetary system. In the case of Jovian stream particles the source regions are Io and the Io plasma torus (Graps et al., 2000), and the particles are composed primarily of condensed KCl and NaCl salts, with traces of sulphates (Postberg et al., 2006). In the case of the Saturnian stream particles the source regions are Saturn's A and E rings (Kempf et al., 2005a), and the stream particles are carbon-rich silicates with traces of more volatile organic species (Kempf et al., 2005b).

Cosmic dust that has impinged upon the Earth has also been investigated in the laboratory (e.g. Jessberger et al., 2001), with the majority of particles found to be chondritic in nature, with anhydrous silicates (pyroxene, olivine for example) and hydrated layer silicates as the most common constituents. Minor constituents in non-chondritic particles are (in order of decreasing occurrence): sulphides, olivine, pyroxene, metal (e.g. Fe/Ni), Calcium-Aluminium rich inclusions (CAIs), carbonates and phosphates. However, it is difficult to determine the origin of dust collected and analysed on Earth, and sources of particular dust particles are usually inferred by comparison with known source features determined through remote

techniques. Regardless of the source of a particular cosmic dust particle, whether it is an ISD or IDP, there is a good probability that it will contain silicates – they are most frequently the constituents of cosmic dust found in the solar system.

In-situ, space-based dust detectors provide the only reliable way to link a particular dust particle to its source (whether a specific body, family of objects or region in space) whilst allowing detailed compositional information to be obtained. Accurate velocity information about the dust particle, prior to its interception (and capture or destruction) by a spacecraft can be achieved in two ways – either close fly-bys of dust-emitting objects of interest, or knowledge of the dust detector’s angular resolution, pointing and velocity, often combined with high accuracy trajectory information from charge detectors.

Auer (2001) provides a thorough historical overview of spacecraft dust detection instruments, so here we will merely summarise recent major developments in the field of “active” (in-situ analysis) dust detection. Advances in both dust capture and in-situ dust analysis techniques have occurred with the successful rendezvous of the Stardust spacecraft with comet Wild/2, Cassini’s tour of the Saturnian system and the launch of the Rosetta spacecraft to rendezvous with comet 67P/Churyumov-Gerasimenko. The Stardust mission included both a dust capture instrument (Brownlee et al., 2003, Brownlee et al., 2006) and also an impact ionisation mass spectrometer, the Cometary and Interstellar Dust Analyser, CIDA (Kissel et al., 2003).

Cassini carries an impact ionisation mass spectrometer, the Cosmic Dust Analyser, CDA (Srama et al., 2004b), whilst Rosetta, yet to reach its target comet, carries a still more complex mass spectrometer, the Cometary Secondary Ion Mass Analyser, COSIMA (Kissel et al., 2007).

Space-based “active” dust detectors often rely on the large speed difference ($\approx \text{km s}^{-1}$) between the dust grains and the detector to obtain compositional information about the dust grains. Intercepted dust is ionised and the resulting ions are analysed using a Time of Flight (TOF) technique to obtain a mass spectrum corresponding to a mixture of charged species (atoms, molecules) originating from both the incoming dust particle and the instrument target material.

Mass spectra produced through impact ionisation instruments (CDA, Kempf et al., 2005a; Hillier et al., 2007a, 2007b; Postberg et al., 2006; Postberg et al., 2008; CIDA, Kissel et al., 2004; PIA (Giotto) and PUMA1 (Vega 1), e.g. Langevin et al., 1987), or in the laboratory (e.g. Stübig, 2002; Goldsworthy et al., 2003) may differ between impacts of similarly composed particles with different masses at different impact speeds. The proportion of atomic and molecular ions generated changes with impact energy and ions that are harder to form may become detectable at higher impact energies. Thus the mass spectrum “fingerprint” from a cosmic dust particle is difficult to interpret (see for example the work by Kissel et al., 1994 and Krueger et al., 2004) without prior calibration of the instrument for a range of particle compositions and impact conditions.

The calibration of impact ionization mass spectrometers usually involves firing analogue particles onto either the flight spare or a laboratory model of the instrument. For high-speed impacts the usual method for accelerating particles to high velocities is by the use of a Van de Graaff accelerator in which dust particles are charged and accelerated through a large potential difference (usually ~ 2 MV). The final velocity attained by dust particles is proportional to the charge to mass ratio of the dust particle – too little charge on too large a

particle and the dust will not electrostatically accelerate. The charge to mass ratio is usually constant for a particular type of dust.

For this method of acceleration to work the particles must therefore be capable of carrying charge and hence the range of materials used has been restricted to those which are either wholly conductive (e.g. iron and copper, Becker & Friichtenicht, 1971; aluminium, Eichhorn, 1976; carbon, Eichhorn, 1976; nickel, Grün et al., 2009; gold, Stübig, 2002; silver, Stübig et al., 2001; tungsten, Eichhorn, 1976; boron carbide, Ratcliff et al., 1997) or those with a conductive coating (organic polymers – latex with a polypyrrole coating, Burchell et al., 1999, 2002; Goldsworthy et al., 2002, 2003; Srama et al., 2004a; polypyrrole-coated alumino-silicate, Goldsworthy et al., 2003; Sulphur-rich latex, Fujii et al., 2006). Recently the successful acceleration of further silicate particles coated with polypyrrole has been reported (Srama, 2007). However the organic coating resulted in low surface charges on the particles, limiting the size range of particles that could be accelerated. The successful acceleration of zinc-coated silicate particles was reported by Göller and Grün (1989). Although their coating method was not reported, the velocity range was lower than that for Fe particles and impact ionization mass spectra were not produced. Later attempts at charging and accelerating gold-coated silicate particles were unsuccessful (Stübig, 2002) with the particles too poorly coated and/or too large.

In general silicates have proved difficult to metal coat without obtaining inhomogeneous coatings or unacceptable levels of agglomeration, and consequently the behaviour of silicate particles during hypervelocity impacts is relatively poorly understood. Research into obtaining mass spectra of silicate particles has tended to use high powered, short duration

190 laser pulses onto a silicate target to simulate the impact of silicate particles, but this method
191 may not accurately reproduce the conditions found in an impact plasma (Sugita et al., 2003),
192 particularly in the case of low velocity impacts (Kadono et al., 2002). Laser pulses are also
193 unsuitable for experiments that specifically require an impacting particle, such as those
194 investigating particle capture by aerogel.

195
196 The primary aim of this research was not to produce a uniform metal coating, or a coating of
197 a specific thickness, but rather to produce particles that were capable of being charged and
198 accelerated through a large potential difference.

199
200 The process described in this paper bonds metal to large areas of the surfaces (“3D” coating)
201 of silicate particles by occurring whilst the particles are in suspension, and reduces
202 agglomeration by frequent ultrasonic agitation of the particles. 0.014 μm fumed silica
203 particles were chosen so that, after a small amount of agglomeration, a wide range of particle
204 sizes would be produced – this range results in a distribution of impact velocities after
205 acceleration in the Van de Graaff accelerator. The material, pure SiO_2 , was chosen as it is
206 easily available and both chemically and physically similar to silicates found in cosmic dust.
207 The coating metal, platinum, was chosen as it fulfils a number of criteria: it has a low cosmic
208 abundance, so it will not bias any results; it is high mass, providing an unambiguous
209 calibration point for mass spectra and ensuring there are no spurious features in
210 mineralogically relevant mass ranges; it is hard, helping to minimise metal-metal sticking of
211 the particles; and it does not occur in any of the space detectors that may use these samples
212 for testing and calibration. From a safety point of view, Pt is inert and reasonably non-toxic,
213 important for storage and handling of the powder.

2. Coating Method

The chemicals were sourced, without exception, from Sigma Aldrich. The experimental method followed, reproduced below, is derived from the methods used in Lee et al., 2007 and Chen et al., 2005, with modifications.

Materials used were: fumed silica (SiO_2 , 0.014 μm diameter), sodium borohydride (NaBH_4), potassium hexachloroplatinate(IV) (K_2PtCl_6), 35 % ammonia solution (NH_4OH), 3-mercaptopropyl trimethoxysilane ($\text{HS}(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$, MPTMS), methanol (CH_3OH , MeOH), isopropanol ($(\text{CH}_3)_2\text{CHOH}$, IPA). The water used throughout the work was Grade 1 water ($> 18.2 \text{ M}\Omega$).

To facilitate the binding of the metal ions to the surface of the fumed SiO_2 , the surface of the SiO_2 was functionalized with thiol-terminated silanising reagent MPTMS. 1 g of fumed SiO_2 was dispersed (stirring and sonication) in 90 mL MeOH, to which was added 0.4 mL of NH_4OH and 3 mL of MPTMS. The stirred suspension was then heated to 60°C for 2 hours, and the MeOH then removed by slow distillation almost to dryness. The concentrated suspension was then washed (by centrifuging and resuspending with sonication) three times with aliquots of IPA to remove unreacted MPTMS.

To prepare for the metallation step, the IPA was solvent exchanged with H_2O . 0.19 g of K_2PtCl_6 , together with 0.5 mL NH_4OH , was then added to the aqueous suspension of functionalized silica particles and stirred at 90°C for 1 hour. Successful binding of the metal salt to the functionalized SiO_2 was demonstrated by the loss of colour from the supernatant

solution. The metallated SiO_2 was then recovered and washed with H_2O . Subsequent reduction of the bound metal salt was achieved via the slow addition of 0.13 g NaBH_4 and 2 mL NH_4OH in 10 mL H_2O to the resuspended SiO_2 (in 90 mL H_2O , initially chilled and slowly heated to 90°C over 30 minutes). The coated SiO_2 was then recovered and washed with H_2O .

The metallation and reduction step was repeated using a further 0.39 g of the metal salt, to increase the amount of metal coating the particles. The resultant metal-coated SiO_2 particles were then recovered, washed with H_2O and dried. The powder was then ball-milled using a stainless steel bearing to reduce further any agglomeration that may have occurred in the drying stage.

3. Results

After metal coating the particles changed from a white, very low-density ‘fluffy’ powder to one that was black and lower in volume. Figures 1 and 2 clearly show the varying size distribution of the coated particles and their aggregate nature. Assuming the original SiO₂ 0.014 µm particles were spherical, and assuming perfect packing, the density of the particles should be a factor of 0.74048 (“Kepler’s Conjecture”) that of the original density of the SiO₂ (2200 kg m⁻³), if the packing is not perfect but “irregular” then the density of the particles should be a factor of ~0.634 (Song et al., 2008) that of the original SiO₂ density. These factors give a density range of ~1393-1626 kg m⁻³. Figure 3, a backscattered electron SEM image of the Pt-coated SiO₂ dust after it has been embedded in resin, cut and polished, also shows the friable agglomerate nature of the particles, as well as localised bright spots corresponding to high Pt concentrations. Previous attempts at coating SiO₂ particles with Pt without using MPTMS also produced these characteristic islands of metal but the resulting particles did not charge. It is therefore likely that in this case there is a thin coating of Pt over much of the particles’ surfaces that is too thin to be visible using backscatter.

Initial tests at the Open University using a low voltage test source indicated that the particles charged successfully, and subsequent tests using the 2 MV Van de Graaff accelerator at the Max Planck Institut für Kernphysik in Heidelberg confirmed that the dust particles not only charged but also could be used as calibration material.

Figures 4, 5 and 6 are examples of cation spectra produced using a laboratory model of the LAMA (Srama et al., 2006; Sternovsky et al., 2007) spectrometer, designed for use in space.

The LAMA spectrometer uses a reflectron to reduce the effect of the ions' initial energies on their arrival times at the detector, producing spectra with a higher mass resolution (>120 , Srama et al., 2006) than instruments such as CDA (10-50; Srama et al., 2004b). These spectra are a sample from nineteen "clean" (i.e. interpretable) spectra generated in the first successfully accelerated batch, chosen as they illustrate the differing features associated with silica in impact ionisation mass spectra, as well as easily identifiable target features, sulphur peaks and the rare case of a Pt feature. The exact masses and velocities of the particles responsible for the spectra shown here are not accurately known but it is possible to derive reasonable limits. The highest velocity recorded during testing with these particles was ~ 30 km s⁻¹ and oxygen usually only appears in spectra at velocities higher than 20 km s⁻¹ (Postberg et al., 2009b) so the impact velocities for these particles probably lie between 20-30 km s⁻¹. Other spectral features (the reduction in the appearance of molecular ions, together with the probable appearance of a weak N⁺ feature) indicate it is likely that the particle velocity increases from Fig. 4 to Fig. 6 and the particle mass thus decreases.

Results from the instrument indicate that particles of >1 μm down to <0.1 μm in diameter, travelling at <4 to >30 km s⁻¹ respectively, produced spectra. The sizes and velocities quoted here are approximate ranges as, in many cases, the particles that produced spectra were too small or too poorly charged to be detected on the accelerator's charge detection unit, from where velocity (and hence mass and size) information is usually gleaned. There were also indications of clusters of particles arriving in bursts, producing garbled spectra due to overlapping mass lines.

4. Discussion

Confirmation that silicate particles have been successfully accelerated and have impacted the instrument can be seen from the peaks at 28-30 u (Si isotopes) and 43.9/44-45 u (SiO). It is also apparent that the platinum coating on the particles is extremely thin, as few traces of Pt (with isotope masses ranging between 190 and 198 u) are seen in the spectra, despite Pt having an ionization energy of only 870 kJ mol^{-1} , whilst many species seen in the spectra have similar, or higher, ionisation energies - e.g. Ag ($731.0 \text{ kJ mol}^{-1}$), Si ($786.5 \text{ kJ mol}^{-1}$), C ($1086.5 \text{ kJ mol}^{-1}$), H ($1312.0 \text{ kJ mol}^{-1}$) and O ($1313.9 \text{ kJ mol}^{-1}$) and, given the impact energies involved, one would expect to see species with lower ionisation energies (Delmann et al., 1977). One of the strongest Pt peaks can be seen in Fig. 4 and may be due to a Pt island such as those shown in Fig. 3. Other features observed in the spectra can be attributed to either the LAMA impact target material (Ag) or common contaminants (organics and the ubiquitous, easily ionised Na and K from perspiration).

It is impossible to say whether (and which of) the contaminants are purely environmental – oil residues from the Van de Graaff accelerator for example, or due to chemicals used during the coating process (S and hydrocarbons from MPTMS, Na from NaBH_4 , K from the Pt salt). It is worth noting that, given the extremely low amount of MPTMS present in the particles (a single monolayer on the surface of the silica), the majority of the organic features in the spectra are probably due to environmental contamination (see, for example, Goldsworthy et al., 2003; Postberg et al., 2009b). The minimization of known organic contaminants is important as large quantities of organics/hydrocarbons make interpretation of the spectra difficult, if not impossible at low velocities, and decrease the likelihood of easy

discrimination between similar but non-identical particle types (organic “interference” from the conducting polypyrrole coating was a problem in interpreting spectra in Goldsworthy et al., 2003). A possible inorganic contaminant can be seen in Fig. 4 at ~103 u (marked with a “?”) – this feature may be due to Rh contamination from other dust coating experiments.

The metal coating process has also been successfully used to coat orthopyroxene [(Mg,Fe)₂Si₂O₆] particles larger in size than the SiO₂ discussed here. The orthopyroxene particles showed signs of charging in a test rig although at the time of writing they have not yet been tested in the Van de Graaff accelerator. Fumed SiO₂ particles have also been coated with rhodium metal, using the hydrated Rh salt RhCl₃(H₂O)₃ instead of the platinum salt. No sonication was performed during the production of Rh-coated SiO₂ particles, resulting in levels of agglomeration too high to produce particles small enough to fly in the Van de Graaff accelerator. Visual examination of the particles indicates that the Rh has been successfully deposited on the surface of the particles, and use of sonication in future should allow Rh-coated grains to be used in hypervelocity impact experiments. Sonication is particularly required when using fumed minerals as starting materials – the extremely small size of the starting particles leads to colloidal behaviour in suspension.

Whilst the technique presented here was developed with hypervelocity impact ionization experiments in mind, the particles are also suited for the calibration of aerogel targets, such as those used on the Stardust spacecraft (Brownlee et al., 2003; Brownlee et al., 2006). The friability of the aggregate particles emulates those of some IDPs (see Jessberger et al., 2001 for example) and enables the behaviour of particles that break up during capture by aerogel (a phenomenon frequently observed in Stardust aerogel targets, Brownlee et al., 2006) to be

better understood. The particles will also be used in studying cratering and residues left on foils.

The process for producing the coated particles still requires considerable refinement – the ability of the coated silicate grains to charge and fly in the accelerator is strongly dependent on the particle mass and the amount of metal on the individual grains. Too much metal and not only do the particles become too heavy to fly, they also undergo too much agglomeration; too little metal and the particles are unable to carry enough charge to allow acceleration in the Van de Graaff, or are too poorly agglomerated and break up during the charging and acceleration process, producing the burst events previously mentioned. Further investigations are planned to quantify the optimum amount of metal to deposit on a given quantity of dust (of a given starting size distribution) to increase the charge carrying potential and tailor the degree of agglomeration.

In summary, the work presented here illustrates a method for successfully producing metal-coated micron and sub-micron cosmic dust analogue particles for use in a Van de Graaff accelerator, without introducing inconvenient levels of contamination, and shows a selection of the initial results from hypervelocity impact experiments.

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Figure captions

Figure 1: An SEM image of the Pt-coated silica particles. The particles were deposited onto gold-plated aluminium stubs using IPA and then, after drying, had a 3-5 nm coating of gold applied.

Figure 2: This image is of a subset of Figure 1 and clearly shows the low-density clusters forming the individual dust particles. The particles were deposited and treated as for those in Figure 1.

Figure 3: A backscattered electron image of the Pt-coated silicate particles. The grains have been embedded in resin, cut and polished.

Figure 4: A LAMA spectrum from the impact of a Pt-coated silica particle onto a silver target. The inset shows the lower mass section of the spectrum. Identifiable abundant species are labelled. The two large features marked with “?” are not yet definitively identified, although the higher mass peak may be due to Rh contamination.

Figure 5: A LAMA spectrum from the impact of a Pt-coated silica grain onto a silver target. The inset shows the lower mass section of the spectrum for clarity. Identifiable abundant species are labelled.

634 Figure 6: A LAMA spectrum from a very small Pt-coated silica particle. The inset shows the
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Figures

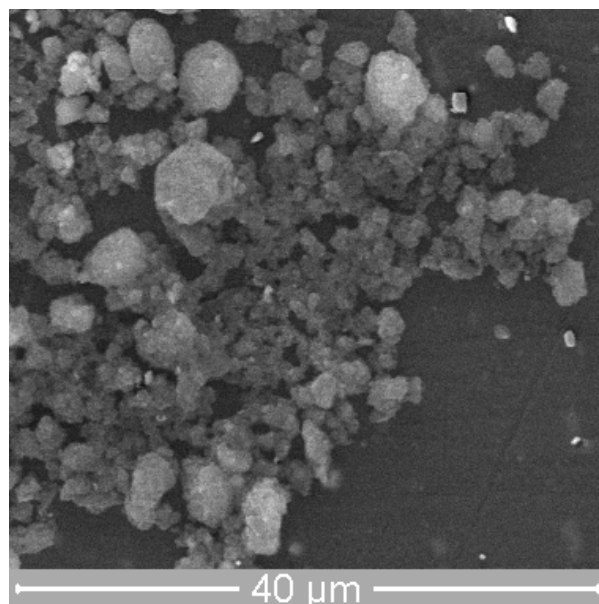


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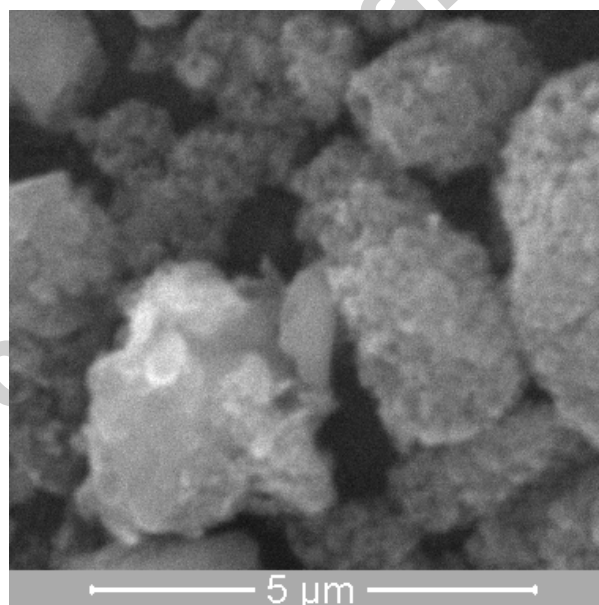


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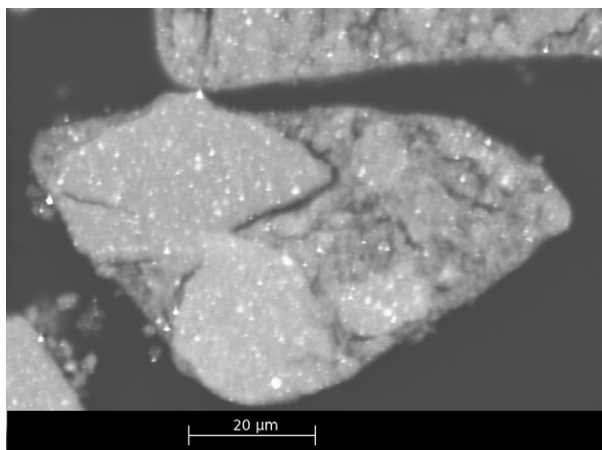


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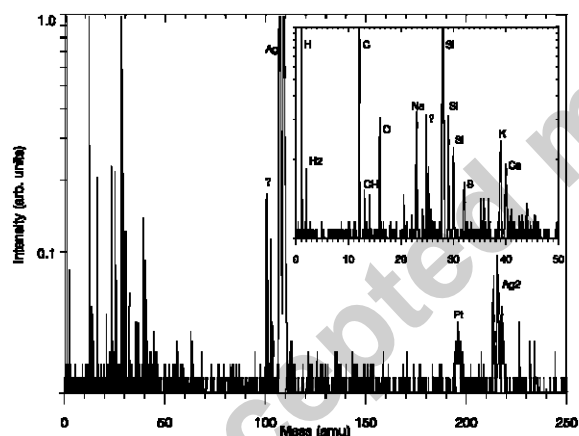


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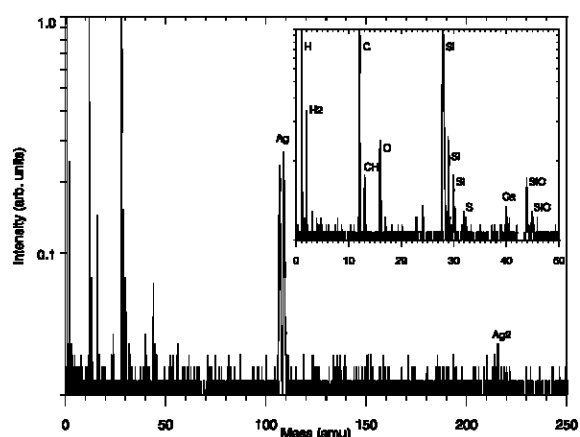


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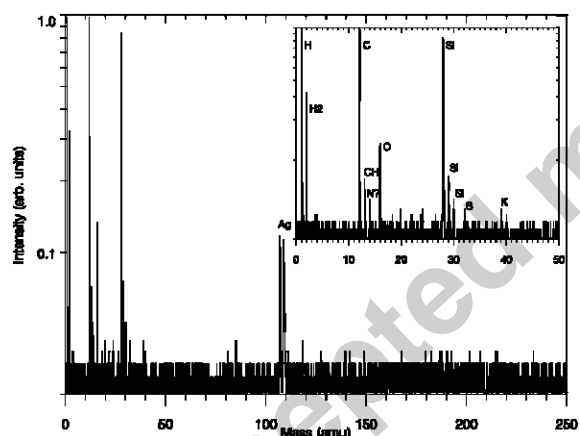


Figure 6: A LAMA spectrum from a very small Pt-coated silica particle. The inset shows the lower mass section of the spectrum for clarity. Identifiable abundant species are labelled.